(REFEREED RESEARCH)

EFFECT OF FIBRE CONTENT ON THE MECHANICAL PROPERTIES OF BASALT FIBRE REINFORCED POLYLACTIC ACID (PLA) COMPOSITES

ELYAF ORANININ BAZALT ELYAF TAKVİYELİ POLİLAKTİK ASİT (PLA) KOMPOZİTLERİN MEKANİK ÖZELLİKLERİ ÜZERİNE ETKİSİ

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ABSTRACT

A widespread use of composite materials leads researchers to search for sustainable raw materials. In the present work, a sustainable thermoplastic polylactic acid (PLA) based composite materials were aimed to produce by extrusion followed with compression moulding method. The chopped basalt fibres with the length of 3 mm were also used as natural inorganic fibre source to reinforce PLA matrices obtained natural resources with the different contents until 50% by weight. Composite pellets were manufactured by a single screw extruder and followed composite panels were manufactured in the hot compression moulding machine. All of the composite samples were experimented to density, tensile, bending and impact tests. The results indicated that the density of the composite materials increase with the increasing of the content of basalt fibres. Besides, the increment of fibre content and about 180% increase compare to pure PLAlinearly enhanced the tensile and flexural modulus of composite materials. As a result of statistical evaluation, basalt fibre reinforcedPLA composites at fibre content of45 wt% were found to have significant difference among other fibre contents and had highest tensile, flexural and impact strength results of 53.73 MPa, 118 MPa and 1.49 kJ/m2, respectively.

Keywords: Basalt fibre, Polylactic acid, Composite, Mechanical properties, Compression moulding

ÖZET

Kompozit malzemelerin yaygın kullanımı araştırmacıları bu alanda kullanılacak sürdürülebilir hammaddeler üzerinde araştırma yapmaya yöneltmektedir. Bu çalışmada, ekstrüzyon ve bunu takiben basınçlı kalıplama yöntemi ile sürdürülebilir termoplastikpolilaktik asit (PLA) matrislikompozit malzemeler üretilmesi amaçlanmıştır. Doğal inorganik bir elyaf kaynağı olarak, 3 mm kırpılmış bazalt elyaflar ağırlıkça % 50 elyaf oranına kadar farklı oranlarda doğal kaynaklardan üretilmiş PLA matrise takviye edilmiştir. Kompozitpellet üretimi için tek vidalı ekstrüder ve ardından sıcak presleme işlemi ile kompozit paneller üretilmiştir. Hazırlanan bütün kompozit numuneler üzerinde yoğunluk, çekme, eğme ve darbe deneyleri yapılmıştır. Sonuçlar, bazalt elyaf takviye oranının artması ile kompozit malzemelerin yoğunluğunun arttığını göstermiştir. Aynı zamanda, artan elyaf oranı ile PLA kompozitlerin çekme, eğilme elastikiyet modüllerinin lineer şekilde artığı ve saf PLA ya göre yaklaşık %180 artış sağlanmıştır. İstatistik değerlendirme sonucunda ağırlıkça %45 elyaf oranında bazalt elyaf içeren PLA kompozitlerinin diğer elyaf oranlarına göre önemli derecede farka sahip olduğu ve sırasıyla 53,73 MPa, 118 MPa ve 1,49 kJ/m2 olmak üzere en yüksek çekme, eğme ve darbe dayanımına sahip olduğu görülmüştür.

Anahtar Kelimeler: Bazalt elyaf, Polilaktik asit, Kompozit, Mekanik özellikler, Basınçlı kalıplama.

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INTRODUCTION

Natural polymers likepolylactic acid, polyglycolic acid, polyhydroxy butyrate and polycaprolactone, have been extensively investigated for plastic and composite applications in order to decrease environmental pollution risk of synthetic plastic wastes. However, these biopolymers are not suitable to use in their native form and need to be converted with chemically, thermally or mechanically in order to achieve technological usefulness [1].

Polylactic acid is among the guide and promising biodegradable polymersand is produced by fermenting of bio based starch sources. Low mechanical properties of PLA matrix need to be improved for many structural applications. Accordingly, PLA matrix were usually reinforced with various type of high strength and high modulus fibres.The use of the natural fibres like jute, kenaf, flax is previously used for PLA matrix reinforcement as anotheruseful choice to the use of the glass fibre in many research studies. Further improvement in performance of these composites can be achieved by inorganicfibres such as carbon and glass or mineral source of basalt fibre[13]. Recently, basalt fibre is used as a good natural substitute to glass fibres because it is obtained from basalt rocks and it was experimentally demonstrated that the fibresof basalt are bioinert and harmless to the human health [2].Basalt fibreare produced as a short and continuous filament by melting of basalt rocks at high temperatures [3].

By improvement of process technology of basalt fibres, a rising generation of basalt fibres show the positive performance features such as noise dampening properties and perfect thermal resistance(superior to glass fibres). Besides that, they are chemical resistant andlow water-logging materials. Due to these reasons, they are proposed to use executions required higher heat resistance. Moreover, the fibres of basalt show high mechanical properties and have low-costs when compared to glass [4]. PLA is basically used for non-structural applications like packaging as a renewable source of polymer it may also take interest for structural engineering applications when modified with fibres and nanoparticles [5, 6, 7, 8].

Neat PLA as a thermoplastic polymer can be processed with extrusion, injection, compression and thermoforming methods.Comparing mechanical properties of composites produced with injection moulding method, results of samples produced with compression moulding method can be obtained to higher stiffness and impact results due to higher fibre loading and fibre lengths. However, heterogeneity of the fibre forming by hand, thereby variability in density distribution and increasing content of porosities in the microstructure can be possible in short fibre reinforced composites by compression moulding. This makes decrease in the strength of the composites[9,10].

The neat PLA have good stiffness of 3 GPa and strength of 60 MPa. However, it has brittle nature failure with low impact energy of about 20 kJ/m^2 and also their heat deflection temperature (50-60°C) is low [11].Bledzki and Faruk investigated mechanical properties of wood fibre reinforced polypropylene composites prepared by injection moulding and compression moulding methods. The results were observed that tensile and flexural strength of injection moulded composites were about 155% and 66% higher than those of compression moulding composites, respectively [12].Even though PLA is mostly reinforced with vegetable fibres for composite manufacturing [13], [14], limited number of study is available with short basalt fibres [15].

Chen et al was first time used basalt fibres in order to improve mechanical properties of pure PLA for hard tissue repair [16]. Kurniavan et. al. compared tensile properties ofneat PLA and untreated basalt fibre/PLA composites. They showed that untreated basalt fibrereinforced PLA readily exhibits reinforcing effects on the composite by having three times the strength and seven times the stiffness of neat PLA [17]. Tabiet. al. investigated the effect of silane treatment on thermal and mechanical properties of short basalt fibre reinforced, extruded and injection moulded PLA composites submitted that properties of silane treated PLA composites were enhanced the performance of the composites. Moreover, the properties of silane treated PLA composites improved with increasing fibre weight ratio up to 40 wt% but the properties of untreated PLA composites were not changed with increased fibre weight fraction [18]. This showed that silane treatment of basalt fibres were positively influenced to properties of PLA composites.

Liu et. al. [19] demonstrated that PLA composites with basalt fibre reinforcement showed superior mechanical properties to those of PLA composites with glass fibre reinforcement. Moreover, they determined that the use of 20wt% basalt fibresconsiderablyenhanced the impact strength of the PLA from 19 kJ/m^2 (unnotchedCharpy) to 34 kJ/m^2 while further fibre addition decreased the impact strength of the composites. Zhang and Xin[20] also investigated the effect of basalt fibre addition on mechanical properties of PLA polymer composites produced by injection moulding. Based on experimental observations, PLA composites with basalt fibre of20vol%resulted best mechanical properties.The aim of this study is to determinethe effect of basalt fibre content on mechanical properties of PLA compositesproduced by extrusion and followed compression moulding method and compare with previous research studies.

2. MATERIAL AND METHOD

2.1 Materials

PLA composites with basalt fibres (BF) reinforcement were manufactured with single extrusion machine followed by pressing consolidation process. Poly-lactic acid (PLA) with the brand of NatureWorks PLA type 3052D was supplied from Oo-kuma Company, Turkey. This type of PLA has a glass transition temperature and melting temperature of 60 °C and 180 °C, respectively. The PLA have a density of 1.24 g/cm^3.Basalt fibres that sized with silane were directly supplied from Spinteks, Turkey. The length of short basalt fibre tows is of 3 mm, the diameter of single fibre is of 15 m and the density of basalt fibres are of 2.63 g/cm^3.

2.2. Composite preparation

Prior to composite production, basalt fibres of 3 mm length at 100 °C for 24 h and PLA granules at 60 °C for 6 h were dried in oven to remove most of the absorbed humidity. The composites were prepared by co-rotating single-screw extruder (RONDOL 3212). In the first step, PLA resin was homogenized with following heat and screw speed parameters. The extruder had three heating zones, and the temperature profiles of the barrel were 190-220-240 °C from the hopper to the die. The screw velocity was set at 75 rpm. The extruded mixture was subsequently cooled down in a water cabin and then long composite strands were pelletized to smaller sizes. Afterwards, the press consolidation technique was applied to prepare the test panels. The panels for test samples were produced under pressure of 45 MPa and the temperature of 190 °C for 5 minutes. After panels were cooled in their mould, the specimens were visually inspected for air bubbles, and those with defects were discarded.

2.3. Composite Testing

The fibre weight fraction was determined by during feeding to extrusion process from 20% to 50%wt fibres with 5%

increase in order to enhance the mechanical performance of the PLA polymers for automobile and construction applications. The basalt fibre weight ratio as a percentage of the PLA composite panelswere codedfrom BF20 to BF50 and compared with neat PLA panels.

The experimental density of the composite samples was determined by using immersion in water method called Archimedesaccording to density standards of ASTM D792. The balance was tarred and the specimen was placed on the weighing pan and weight of the specimen in air was recorded as M_{air}. The specimen was placed onto the weighing basket, the samples were submerged, and the weight was recorded as $M_{submerged}$. The Archimedes density or experimental density of the samples was calculated using the following equation:

$$Density\ of\ solid\ =\ [\frac{(M_{air})}{M_{air}-M_{sebmerged}}]\ x\ density\ of\ water$$

The tensile testing (ASTM D638), 3-point bending testing (ASTM D790) were carried out by universal testing machine (MTS criterion model 45, made in USA). An impact testing that is type of Charpyunnotched were carried out with Instron model (Ceast 9050, made in Germany) impact pendulum machine.For each mechanical test was done with different samples that have same components for 5 times and the average values and ± standard deviation was demonstrated in the tables. The analyse of variance (ANOVA) with the Post-Hoc test was also used to test significant difference between test groups in terms of tensile, flexural and impact properties of BF/PLA composites. The sample sizes were cut according to the ASTM standards.Finally, the fracture surfaces of the specimens were observed using a scanning electron microscope (SEM) (ZEISS EVO LS10, Germany) with a field emission gun and an accelerating voltage of 5Kv. the samples were dried at 50 $\pm$ 3^{o}C for 48 h before SEM studies. Morphology of the fractured composites after tensile testing was Figure 17. A gold layer of a few nanometres in thickness was coated onto the fracture surfaces. The samples were scanned perpendicular to the fractured surface. SEM micrographs were taken at the magnification level of 250x.

RESULTS AND DISCUSSIONS

The results of the experimental density of the samples were presented in Table 1. It is shown that the addition of the basalt fibres increased the density of the PLA panels. The highest ratio of 50wt% of basalt fibres showed density value of 1.63 g/cm^3 and 25% higher value than that of neat PLA panels. The theoretical value of fibre volume fraction of the basalt fibre composites were ranged between 11% and 32%.

Stress strain behaviour of BF/PLA composites can be seen from the Fig. 1. It is observed that both the neat PLA and basalt fibres reinforced PLA composites showed viscoelastic behaviour. Addition of basalt fibres lead to increase significant rise in stiffness of PLA polymer.

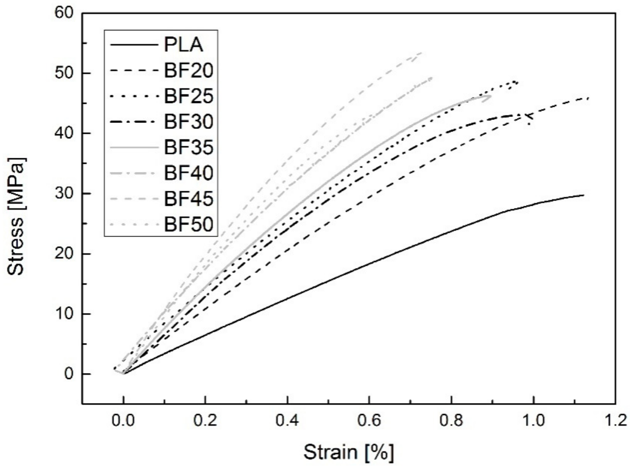

Figure 1. Stress-strain behaviour of PLA composites with basalt fibre reinforcement in different basalt fibre contents

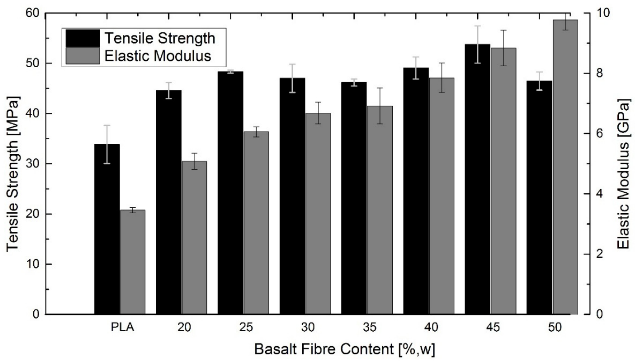

Figure 2. Tensile strength and elastic modulus of BF/PLA composites based on various fibre contents.

Table 1. Theoretical fibre weight, fibre volume fraction and density of the neat PLA and basalt fibre reinforced PLA composites

Sample No	Fibre Weight Fraction	Fibre Volume Fraction	**Density** [g/cm^3]
PLA	0	0	1,23 ± 0,01
BF20	20	11	1,37 ± 0,02
BF25	25	14	1,39 ± 0,02
BF30	30	17	1,43 ± 0,02
BF35	35	21	1,45 ± 0,02
BF40	40	24	1,52 ± 0.03
BF45	45	28	1,58 ± 0,02
BF50	50	32	1,63 ± 0,02

Figure 2 shows tensile strength and modulus of BF/PLA composites in different fibre contents. The tensile strength was increased until fibre content of 45wt% and then decreased again at fibre content of 50wt%.The tensile modulus of composites was gradually increased with increased fibre content. The basalt fibre improve the tensile strength of fibre composites from 45 MPa to 54 MPa by increased fibre content from 20 wt% to 45 wt%. It is seen that the tensile strength results at lower fibre ratios (from 20 to 35 wt%) does not show any significant difference but at fibre content of 40 and 45 wt% tensile strength results significantly increased. Meanwhile tensile modulus results of composites were linear relationship with fibre content. The basalt fibres effectively changed modulus values of PLA composites with increased fibre content. The modulus results of 3 GPa to 9 GPa by increased fibre content from 20wt% to 45 wt%

Figure 3 shows flexural strength and modulus of BF/PLA composites with increasing fibre content from 20 to 50 wt%. Besides, the addition of basalt fibres from 20wt% to 50wt% did not show any significant increase in tensile strength values compared to value of 102 MPa for neat PLA polymer. However, adding ofbasalt fibresfrom 20wt% to 50 wt% increase gradually flexural modulus values from 4.65 GPa to 9.18 GPa.

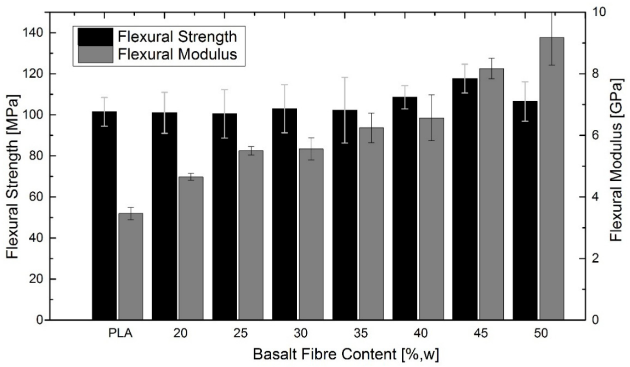

Figure 3. Flexural strength and flexural modulus of BF/PLA composites with various basalt fibre contents.

Both mechanical test results clearly showed that tensile and flexural modulus of neat PLA polymer was improved by basalt fibre contribution. Because, the modulus of the composites mainly depends on fibre content and fibre modulus, orientation and length [22].However, tensile strength results are more sensitive to show remarkable change compared to flexural strength by addition of basalt fibres. This might be due to the fact that short fibre length does not show significant effect on flexural testing.

All tensile flexural and impact testing results of basalt fibre reinforced PLA composites at different fibre weight ratios are presented in Table 2 for comparison. The results of statistical analysis of each test data with the letter of indices were also summarised.

The one way Anova test results indicated significant difference among test factors and Duncan homogeneity test were employed to compare the significant difference between the mean of each group at a 95% confidence level. The different groups showed with a,b,c,d,e letters as shown above.The highest tensile and flexural strength were obtained in the 45%wt basalt fibre composites (53.73 MPa, 118MPa) for basalt fibre reinforced PLA composites, while the highest tensile and flexural modulus in the composites were obtained in fibre weight ratio of 50wt% (9.77 GPa, 9.18 MPa). Pure PLA polymer showed significantly lower tensile strength, lower flexural strength with the values of 33.84 MPa, 101.5 MPa respectively comparing composite samples.

When the impact properties examined, it was determined that basaltfibre significantly increased unnotchedCharpy impact strength of the PLA polymers. By adding 45wt% of basalt fibres, the impact strength showedthe value of 18.3 kJ/$m^2$with the increase of 165% from the original value of neat PLA of 6.9 kJ/m^2. When basalt used at higher than 45wt%, the impact strength of the composites was decreased to 16.4 kJ/m^2.

According to the previous research studies, the mechanical performance of chopped basalt fibre reinforced thermoplastic composites which are processed with different methods, fibre content and matrices are summarized and compared with the optimum results achieved in this study in Table 3. It is shown that similar findings with this study were observed by other researchers for basalt fibre reinforced PLA composites [18-21]. However, in this study, tensile and flexural strength values showed significantly improvement on PLA composites. In a previous study [21], the basalt fibre reinforced PLA composites at optimum 40 wt% shows tensile strength of 110 MPa by injectionmoulding. In another study, BF/PLA composites by injection moulding showed tensile strength of 120 MPa at 45wt% in optimum fibre

Table 2. Thetensile,flexural and impact properties of with basalt fibre reinforced PLA composites

Sample name	Tensile Strength [MPa]	Tensile Modulus [GPa]	Strain to Failure [%]	Flexural Strength [MPa]	Flexural Modulus [GPa]	Impact Strength [kJ/m^2]
PLA	**33.84**^{a}± 3.8	**3.46**^{a}± 0.09	**1.22**^{de}± 0.06	**102**^{a}± 7	**3,46**^{a}± 0.20	**6.9**^{a}± 1,3
BF20	**44.59**^{b}± 1.6	**5.08**^{b}± 0.27	**1.18**^{de}± 0.06	**101**^{a}± 10	**4,65**^{b}± 0.11	**13**^{b}± 2.5
BF25	**48.34**^{bc}± 0.3	**6.06**^{c}± 0.17	**1.04**^{d}± 0.05	**101**^{a}± 12	**5,50**^{c}± 0.14	**14**^{bc}± 2.5
BF30	**47.00**^{bc} **±** 2.8	**6.68**^{cd}± 0.36	**0.97**^{c}± 0.06	**103**^{a}± 12	**5,56**^{c}± 0.36	**14.6**^{bc}± 1
BF35	**46.18**^{b}± 0.7	**6.92**^{cd}± 0.6	**0.84**^{b}± 0.05	**102**^{a}± 16	**6,25**^{d}± 0.48	**14.8**^{bc}± 1.7
BF40	**49.06**^{bc}± 2.2	**7.85**^{e}± 0.49	**0.83**^{b}± 0.07	**109**^{ab}± 6	**6,57**^{d} ± 0.74	**18.3**^{d}± 2.3
BF45	**53.73**^{d}± 3.7	**8.84**^{f}± 0.59	**0.72**^{b}± 0.06	**118**^{b}± 7	**8,17**^{e}± 0.33	**18.5**^{d}± 2.3
BF50	**46.49**^{bc}± 1.8	**9.77**^{g}± 0.33	**0.58**^{a}± 0.08	**107**^{ab}± 10	**9.18**^{f}± 0.90	**16.4**^{c}± 2.2

content [19]. This showed that compression moulded BF/PLA composites showed significantly lower tensile strength compared to injection moulded composites. Another factor in terms of strength results, fibre length can be applied at longer sizes by injection moulding compare to those of compression moulding method [23]. Because the fibre pellets were milled to smaller fibre sizes for homogenous density distribution on compression and this usually performed at the significantly lower level of critical fibre length which is important for achieving higher strength values [24]. Bledzki and Faruk also expressed that addition of compabilitizers and mixing of polar and nonpolar components can be provided by injection moulding comparing compression moulding process[12].

Figure 4 shows the fractured surfaces of basalt fibrecontent of 20% and 45%wt in PLAcomposites based on the fracture surface of the tensile failed samples.It is clearly seen that there was perfect adhesion situated between the PLA and the basalt fibres at 45wt of basalt fibre contentwhich is shownwith the disappearance of any gap at the root of the PLA and the fibre.However,SEM observation of 20%wt of the samples indicated the poor interfacial adhesion basalt fibres with PLA matrix. Small broken fibres and holes can be observed in the composite fracture surfaces due to pull out individual fibres on both fracture surfaces. However, the number of broken and pull out fibres were increased and clearly observed in samplefracture surfacesof the fibre ratio of 45 wt%. More homogenously distributed fibresare also seen onfracture surfaces ofsamples with increasingfibre content compared to fibre and matrix rich zones atlow fibre ratios. Fracture surface of the all composites show that the distribution of the fibres is considered to be some degree of anisotropy rather than to be in random distribution due to the fact that the processing factors such as pressure and extrusion directions.

CONCLUSIONS

In the present study, biodegradable PLA polymerwas reinforced in different basalt fibreweigh fractionsto improve mechanical properties of the PLA plastics for engineering applications. Six different fibre content up to 50 wt% of basalt fibreswere successfully compounded with PLA matrix by conventional extrusion and compression moulding processing method in succession. The study proved that significant improvement in tensile, flexural and impact strength of the composites was observed as the weight percentage of basalt fibre. The similar result shown below can be concluded:

- Fibre content is having significant effect on mechanical performance of the PLA composites with chopped basalt fibre reinforcement.
- The optimum tensile and flexural and impactproperties of the PLA composites were achieved at45wt% basalt fibre content by compression moulding of extruded composite pellets.
- Increasing fibre weight fraction up to 50wt%results to increase linearly the density, tensile and flexural modulus of the composite materials.

Table 3. Mechanical properties of basalt fibre reinforced composites in previous research studies.

Material	Method	Fibre content	Tensile strength (MPa)	Elastic modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact Strength (kJ/m^{2})	Reference
Basalt-PBS	Injection	15 vol%	45	1.1	70	3.75	7.5	[6]
Basalt- PP	Injection	20 vol%	30	2.3	64	3.2	65	[20]
Basalt-PLA	Injection	27 wt%	120	6	190	9	65	[19]
Basalt-PLA	Injection	40 wt%	110	-	145	-	20	[21]
Basalt-PLA	Injection	40 wt%	120	8	180	12	40	[18]
Basalt-PLA	**Compression**	**45 wt%**	**54**	**8.8**	**118**	**8.2**	**18.5**	**This study**

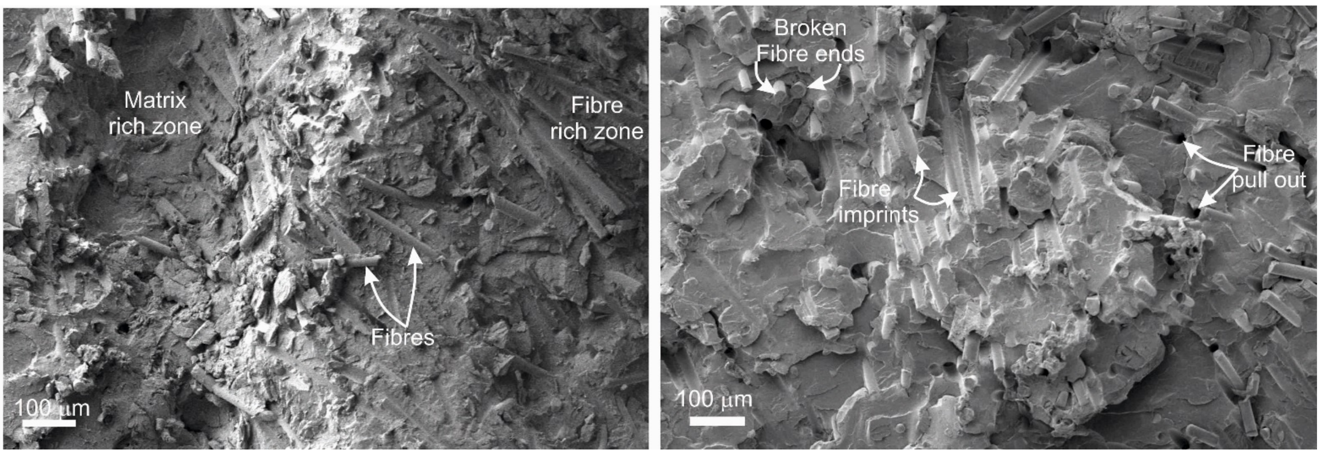

Figure 4. SEM images of basalt fibre reinforced composites with fibre ratio of 25wt%and 45wt%

- The impact energy value of the basalt fibre reinforced composites also found about 2 times higher compared to that of neat PLA polymer.
- Fibre breakage were dominant in the fracture surfaces of the basalt PLA composites.

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